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L4: Entry 2 of 18 File: USPT Feb 12, 2002

DOCUMENT-IDENTIFIER: US 6346195 B1
**** See image for Certificate of Correction ****
TITLE: Ion exchange removal of metal ions from wastewater

Abstract Text (1):
A novel process and apparatus are disclosed for cleaning wastewater containing metal ions in solution, hydrogen peroxide, and high solids, e.g., greater than about 50 mg/l particulate solids. A carbon adsorption column removes hydrogen peroxide in the wastewater feed containing high solids. A ion exchange unit removes the metal ions from solution. The process and apparatus remove metal ions such as copper from a high solids byproduct polishing slurry from the chemical mechanical polishing (CMP) of integrated circuit microchips to form an environmentally clean wastewater discharge.

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Brief Summary Text (11):
The chemical mechanical polishing (CMP) planarization process involves a polishing slurry composed of an oxidant, an abrasive, complexing agents, and other additives. The polishing slurry is used with a polishing pad to remove excess copper from the wafer. Silicon, copper, and various trace metals are removed from the silicon structure via a chemical/mechanical slurry. The chemical/mechanical slurry is introduced to the silicon wafer on a planarization table in conjunction with polishing pads. Oxidizing agents and etching solutions are introduced to control the removal of material. Deionized water rinses often are employed to remove debris from the wafer. Ultrapure water (UPW) from reverse osmosis (RO) and demineralized water also can be used in the semiconductor fabrication facility tool to rinse the silicon wafer.

Brief Summary Text (13):
The chemical mechanical polishing (CMP) planarization process introduces copper into the process water, and governmental regulatory agencies are writing regulations for the discharge of wastewater from the chemical mechanical polishing (CMP) planarization process as stringently as the wastewater from an electroplating process, even though CMP planarization is not an electroplating process.

Brief Summary Text (14):
The copper ions in solution in the waste-water must be removed from the byproduct polishing slurry for acceptable waste-water disposal.

Brief Summary Text (22):
Ion exchange technology is effective for concentrating and removing low levels of contaminants from large quantities of water. Ion exchange also has been employed effectively in wastewater treatment for removal of specific contaminants. For ion exchange to remove specific contaminants from wastewater economically, it is often important to utilize a selective resin or create an ionic selectivity for the

specific ion that has to be removed.

Brief Summary Text (23):
Many ion exchange resin manufacturers developed selective resins during the 1980's. These ion exchange resins received wide acceptance because of their high capacity and high selectivity over conventional cation and anion resins for certain ions.

Brief Summary Text (24):
Cation selective resins have demonstrated their ability to remove transition metals from solutions containing complexing agents such as gluconates, citrates, tartrates, and ammonia, and some weak chelating compounds. These selective resins are called chelating resins, whereby the ion exchange sites grab onto and attach the transition metal. The chelating resin breaks the chemical bond between the complexing agent or a weaker chelating chemical.

Brief Summary Text (25):
The conventional cation resins have a much greater difficulty in removing specific metals from waste streams that are chelated or contain complexing agents. The conventional resins exhibit low or no capacity for removing heavy metals in the presence of complexing or chelating compounds.

Brief Summary Text (26):
The ion exchange resin is used to pull the copper ions out of solution.

Brief Summary Text (27):
Brown, U.S. Pat. No. 4,666,683; Etzel et al., U.S. Pat. No. 4,210,530; Merchant, U.S. Pat. No. 4,329,210; and Gefart, U.S. Pat. No. 5,256,187 disclose removing copper by ion exchange.

Brief Summary Text (28):
If hydrogen peroxide (H.sub.2 O.sub.2) is present, the ion exchange resin will be oxidized, and the resin structure is broken down. Accordingly, hydrogen peroxide can not be present in an ion exchange unit operation because the ion exchange resin is incompatible with hydrogen.

Brief Summary Text (31):
Asano et al., U.S. Pat. No. 3,923,741, in Example 3 pass a copper solution through a granular active carbon column. Flow resistance is measured and reported. The solution then is passed through an ion exchange resin column. (U.S. Pat. No. 3,923,741, Col. 6, lines 35-65.)

Brief Summary Text (32):
U.S. Pat. Nos. 5,476,883, 3,923,741, and 3,941,837 teach precipitating copper ions in wastewater solutions using a carbon column and ion resin exchange beds. In U.S. Pat. No. 5,476,883, copper is removed by strongly acidic cation exchange resin. (Col. 11, lines 36-52.) Example 8 sets up a Calgon CPG coal-based activated carbon column followed by ion exchange resin. (Col. 12, lines 55-67.) Peroxide concentrations are disclosed in Table 2.

Brief Summary Text (33):
Ion exchange can be used to attach copper ions, but would not be likely to work on a byproduct polishing slurry because of the quantities of solids coming in with the byproduct polishing slurry in the form of a silica, alumina slurry.

Brief Summary Text (35):
Ion exchange resin suppliers and equipment manufacturers strongly advise that particle controls ahead of, i.e., upstream from, ion exchange bed systems are an essential aspect of an effective pretreatment system.

Brief Summary Text (36):

According to Bayer, the feed to the ion exchange resin bed should be as free as possible of suspended solids.

Brief Summary Text (37):
Particles of suspended solids bind up the ion exchange resin beds. The resin acts as a filter to retain the particles. The suspended solids accumulate and cause an increase in pressure drop across the resin bed. When this increased pressure drop occurs, the water is forced to take the path of least resistance and circumvents or flows around the resin bed. This resin circumvention is called channeling. When the process water flows down the sides of the column, a large portion of the resin is bypassed, limiting the contact between the resin and the process water, resulting in high contaminant leakage and poor bed capacity. Under extreme conditions, internal distributors and collectors can break due to the high pressure drop.

Brief Summary Text (38):
An ion exchange bed that is loaded with solids is difficult to regenerate. The regenerant solution takes the path of least resistance and channels down the sides of the column resulting in incomplete regeneration of the resin.

Brief Summary Text (39):
According to Rohm and Haas, the feed must be relatively free from suspended solids and colloidal material. The suspended solids and colloidal matter will form a mat at the surface of the bed. Pressure drop increases, channeling is encountered, and portions of the bed are by-passed. The suspended solids and colloidal matter also coat the beads and granules of the ion exchange resin, reducing the rate of diffusion of ions in and out of the exchanger resin. It is therefore important that all feeds be clarified as much as possible to remove the last traces of suspended solids or colloidal matter. Coagulation, sedimentation, and filtration are the normal clarifying methods.

Brief Summary Text (40):
The byproduct polishing slurry wastewater containing copper ions from the CMP of semiconductor microelectronic chips containing copper can be passed through a microfilter to remove the solids in the form of the silica, alumina slurry. The permeate from the microfilter containing permeate copper ions then can be contacted with the ion exchange resin to attach the copper.

Brief Summary Text (57):
The process and apparatus of the present invention remove metal ions from wastewater by providing a first step carbon adsorption bed for receiving a wastewater feed containing metal ions in solution, wherein the wastewater feed contains solids sized in the range of about 0.01-1.0 .mu.m in an amount higher than about 50 mg/l, in combination with providing a second step ion exchange unit operation for receiving a carbon bed product stream from the carbon adsorption bed and for removing the metal ions from solution. The process and apparatus of the present invention remove metal ions from wastewater containing solids in an amount higher than about 100 mg/l, e.g., by way of example in an amount in the range of about 500-2000 mg/l.

Brief Summary Text (59):
The ion exchange unit operation includes means for contacting copper ions in the carbon bed product stream with a chelating ion exchange resin to attach the copper ions. In one aspect, the chelating ion exchange resin includes a macroporous iminodiacetic functional group. In one embodiment, the chelating ion exchange resin includes a high degree of cross-linked poly-styrene resin which provides for superior chemical resistance to oxidizers.

Brief Summary Text (60):
The process and apparatus of the present invention operate to remove metal ions from a wastewater from a byproduct polishing slurry. In one embodiment, the process

and apparatus of the present invention operate to remove metal ions, e.g., such as copper metal ions, from a wastewater from a byproduct polishing slurry from the chemical mechanical polishing (CMP) of integrated circuit microchips to attach the metal ions and form an environmentally clean water discharge product.

Detailed Description Text (2):
The process and apparatus of the present invention provide for a removal of metal ions through a combination of steps including passing a wastewater solution containing metal ions first through a carbon adsorption column, preferably without prior micro-filtration or ultra-filtration removal of suspended solids, to remove hydrogen peroxide (H.sub.2 O.sub.2) catalytically and then contacting the wastewater solution containing metal ions with an ion exchange resin to remove the metal ions from solution.

Detailed Description Text (4):
In one aspect, the process and apparatus of the present invention provide a novel process and apparatus for the removal of copper ions including passing a wastewater solution containing copper ions first through a carbon column, preferably without prior micro-filtration or ultra-filtration removal of silica, alumina slurry solids, to remove the hydrogen peroxide (H.sub.2 O.sub.2) catalytically and then contacting the wastewater solution containing copper ions with a chelating ion exchange resin to attach the copper.

Detailed Description Text (5):
In one embodiment, the process and apparatus of the present invention provide a novel apparatus and process for the removal of copper ions including passing a wastewater solution containing copper ions first through a carbon adsorption column, preferably without prior micro-filtration or ultra-filtration removal of silica, alumina slurry solids, to remove catalytically the hydrogen peroxide (H.sub.2 O.sub.2) and then contacting the wastewater solution containing copper ions with a chelating ion exchange resin of iminodiacetic functional group to attach the copper.

Detailed Description Text (9):
The collection tank 20 has a nominal retention time of 10 minutes at an influent flow rate of 10 gpm. The copper CMP wastewater contains oxidizers, dissolved copper, copper etchants, alumina particles, silica particles, and some times a corrosion inhibitor. These copper CMP wastewater constituents are contained in a background of deionized (DI) water. The following constituents are common, including dissolved copper, total suspended solids, oxidizing agents, etchants, complexing agents, DI water background 99% +, TDS 800, pH 6 to 7.

Detailed Description Text (10):
The copper CMP wastewater passes from the collection tank 20 in line 22 and line 24 to a pump 30 and a pump 40, respectively. The pumps 30 and 40 provide a duplex pumping station for transferring the wastewater through lines 32 and 42, respectively, at pressures indicated by pressure gauges 32 and 44, through throttling valves 36 and 46, and through lines 38 and 48 to main line 50 at a flow rate of 15 gpm. Five gallons per minute pass in line 52 and are diverted back to the collection tank 20. The remainder of the waste stream (10 gpm) is directed to the process equipment. The 5 gpm of recirculated water maintains a high velocity in the feed tank collection tank 20, which minimizes solids accumulation.

Detailed Description Text (11):
The recirculated water in line 52 can also receive an injection of mineral acid if the CMP tool is shut down for an extended period of time. A proportional pH meter maintains the pH of the wastewater during these excursions. Normal operation of the Copper removal/recovery system does not require pH control.

Detailed Description Text (13):

The wastewater stream in line 50 is passed in line 54, provided by a flexible hose made of a material such as stainless steel nylabraid, to the top of a carbon column 60. The wastewater in line 54 flows at 10 gpm to a peroxide removal pretreatment in carbon column 60. The carbon column 60 contains a specialized coarse grade of activated carbon. The activated carbon is used to remove the hydrogen peroxide. The peroxide pretreatment column is replaced every three months with a fresh column. The carbon column 60 contains granular activated carbon particles sized in the range of about 8.times.40 mesh. A suitable carbon is 8.times.30 mesh acid washed available from U.S. Filter Westates Carbon--Arizona Inc. in Parker, Ariz. The hydrogen peroxide of the wastewater stream 54 passes down-flow in the carbon column 60 and is adsorbed onto the granular activated carbon in the carbon column 60.

Detailed Description Text (14):
Referring now to FIG. 3, a rear elevational process equipment schematic is shown of the process and apparatus of the present invention. Following the carbon bed treatment step in carbon column 60, a carbon column product stream 62 containing copper ions in solution and grinding (polishing) solids from the carbon column 60 is passed to an ion exchange copper scavenging column 70. The ion exchange copper scavenging column 70 contains a specialized copper scavenging resin that features particle grading that is used to control the resin bead size and maintain a minimum uniformity coefficient.

Detailed Description Text (15):
The specialized resin system removes the copper and allows the particulate to pass through to the discharge. The resin system features two ion exchange columns 70 and 80 in series. A worker (ion exchange column 70), polisher (ion exchange column 80) arrangement allows for maximum loading of the copper on the resin.

Detailed Description Text (16):
The primary or worker ion exchange resin ion exchange column 70 is replaced every 17 to 18 days of continuous operation. The lag or polisher ion exchange column 80 is placed in the primary position, and a freshly regenerated column is placed in the polishing position. The spent ion exchange column is conditioned and regenerated off site at a licensed service facility provided by U.S. Filter Wastewater Group, Inc. of Warrendale, Pa.

Detailed Description Text (17):
A RCRA part B facility is utilized for resin regeneration in the event that the resin is considered a hazardous waste.

Detailed Description Text (18):
Environmentally clean wastewater slurry passes through wastewater ion exchange bed discharge 90 to a municipal drain.

Detailed Description Text (19):
The copper is removed from the resin during the conditioning and regeneration procedure. The copper is then recovered as elemental copper and re-sold to the wood preservative or metals industry.

Detailed Description Text (21):
The product water from CRS enters the SSRS collection tank where the waste stream receives an injection of a chemical coagulant. The coagulant feed is optional. The coagulant is used to greatly enhance the recovery rate of the subsequent microfilter. If, however, the intention is to discharge the copper free solids system for subsequent discharge to the Publicly Owned Treatment Works (POTW), then a high microfilter recovery is not desirable.

Detailed Description Text (23):
If it is desirable to eliminate TSS in the reject from the microfilter, a chemical coagulant is utilized. Again the coagulant has the effect of greatly improving the

microfilter recovery rate and increasing the flux. The concentrated, copper-free solids are fed to a filter press for de-watering. A recovery rate greater than 95% is achievable. The remaining 5% microfilter blow down is easily de-watered in the filter press.

Detailed Description Text (24):
The product water from the microfilter is received in a collection tank. The tank serves as a break tank and supplies water to the RO feed pumps. The RO feed pumps provide the feed pressure required for the reverse osmosis system.

Detailed Description Text (25):
The re-pressurized feed water to the RO unit enters a bank of RO membranes. The RO membranes are used to remove dissolved solids to prepare the water for reclaiming in the fabrication facility.

Detailed Description Text (30):
Copper CMP wastewater contains oxidizers, dissolved copper, copper etchants, alumina particles, silica particles and sometimes a corrosion inhibitor. These constituents are contained in a background of deionized water. The following constituent concentrations are common.

Detailed Description Text (32):
When the resin is laden with copper, the resin is regenerated with mineral acid to remove the copper. It is important to establish a reversible reaction and remove the copper from the spent resin. Replacement cost of chelating resin to be supplied for each loading cycle would be prohibitive.

Detailed Description Text (33):
Sulfuric acid is the acid of choice for conventional resin regeneration. Sulfuric acid is less costly than other mineral acids such as hydrochloric or nitric acid. Another major benefit from sulfuric acid regeneration resides in the ability to easily electrowin the copper from the spent acid following regeneration of the chelating resin. The elemental copper from the electro-winning process could then be sold as scrap metal with a minimal environmental liability.

Detailed Description Text (34):
It has been found empirically that the ion exchange resin in the process and apparatus of the present invention preferably is not regenerated with sulfuric acid. It has been found empirically that the combination of alumina, silica, and sulfate in the process and apparatus of the present invention become cemented together.

Detailed Description Text (35):
The resin column in the process and apparatus of the present invention was practically solidified during regeneration with sulfuric acid.

Detailed Description Text (36):
It has been found empirically that the ion exchange resin in the process and apparatus of the present invention preferably is regenerated with hydrochloric acid.

Detailed Description Text (37):
The ion exchange resin in the process and apparatus of the present invention preferably is regenerated with hydrochloric acid, even though it is problematic to electrowin copper from the hydrochloric acid regenerant solution, and even though chlorine gas is a by-product from this electrowinning operation.

Detailed Description Text (38):
It has been found that a service-based ion exchange process and apparatus of the present invention is preferred for removing copper from CMP slurry from the

manufacture of microchips. The service-based ion exchange process and apparatus of the present invention have been found to recover the copper with specialized recovery technology HTMR (High Temperature Metal Recovery). The service-based ion exchange process and apparatus of the present invention have been found to recover the hydrochloride regenerant solution with distillation.

Detailed Description Text (39):
The combination of silica particles, alumina particles, and sulfates has been found to be a problem in regeneration of the ion exchange resin when using sulfuric acid.

Detailed Description Text (40):
In anticipation of difficult electrowinning operations in a field application using hydrochloric acid, and to provide a minimum liability to the microchip manufacturer, a service-based process and apparatus are preferred. The preferred method provides for the recovery of the ion exchange resin loaded with copper, provides for the recovery of copper, and provides for the recovery of hydrochloric acid regenerant.

Detailed Description Text (41):
The resin is regenerated at the TSDF (Treatment Storage and Disposal Facility) using recovered hydrochloric acid. The copper chloride then is sold as a by-product, and the hydrochloric acid is recovered in an acid recovery unit operation. Alternatively, the copper is precipitated as a metal hydroxide and then recovered in an HTMR High Temperature Metal Recovery operation as elemental copper.

Detailed Description Text (42):
The ion exchange resin of the process and apparatus of the present invention preferably is a chelating ion exchange resin. The chelating ion exchange resin of the process and apparatus of the present invention preferably has a high degree of cross-linking. The cross-linking increases chemical resistance to oxidizers. The chelating ion exchange resins of the process and apparatus of the present invention can isolate the copper from complexing agents and most chemical chelants. High concentrations of oxidizers such as hydrogen peroxide need to be removed in a pretreatment step.

Detailed Description Text (43):
The chelating ion exchange resins of the process and apparatus of the present invention have an operating capacity in the range of about 1.5 to 2.0 pounds/ft.sup.3 of copper. A minimum operating capacity for the chelating resin is about 1.5 pounds/ft.sup.3 of copper.

Detailed Description Text (44):
Copper can be taken up by the resin in the process and apparatus of the present invention at a low pH. It has been found that the process and apparatus of the present invention preferably is positioned on-site at the copper CMP tool facility to avoid aging of the copper CMP wastewater slurry containing copper. In the event of such an on-site process and apparatus of the present invention, the pH may be maintained near neutral. For aged copper CMP wastewater slurry feed solution, a pH of about 2 to 3 provides a good copper uptake on the resin in the process and apparatus of the present invention.

Detailed Description Text (45):
The chelating ion exchange resin of the process and apparatus of the present invention provides a tight uniformity coefficient of 1.7 maximum. The ion exchange resin of the process and apparatus of the present invention is screened to control bead size. Bead size control is necessary to minimize suspended solids build up in the bed. The ion exchange resin of the process and apparatus of the present invention has the following minimum specifications.

Detailed Description Text (46):
The ion exchange resin of the process and apparatus of the present invention provides a controlled bead size because of a sieving process. A tight control of bead size eliminates undesirably small beads and resin fines which could eventually trap suspended solids.

Detailed Description Text (47):
The ion exchange resin of the process and apparatus of the present invention provides a higher operating capacity of the iminodiacetic resins evaluated.

Detailed Description Text (48):
The ion exchange resin of the process and apparatus of the present invention is manufactured by the thalamid functionalization process. The thalamid functionalization process reduces the resin's environmental toxicity. It is preferred not to use chloromethylene to functionalize the groups of the ion exchange resin of the process and apparatus of the present invention. Chloromethylene has been labeled as a carcinogen.

Detailed Description Text (55):
A novel method and apparatus provided a first step carbon adsorption removal of hydrogen peroxide from a wafer CMP planarization polishing waste combined with a second step using ion exchange to remove complexed copper in the wafer planarization polishing waste. The wafer planarization polishing waste contained many particulate alumina solids which otherwise, i.e., if not for the copper, could be disposed via a municipal drain or sewer.

Detailed Description Text (58):
The carbon used during all carbon column testing was Calgon RX 8.times.40 mesh (Lot 04033) available from Calgon Carbon Co. in Pittsburgh, Pa. A suitable equivalent carbon is 8.times.30 mesh acid washed available from U.S. Filter Westates Carbon--Arizona Inc in Parker Ariz. The carbon was prepared by degassing and rinsing. Prior to the experimental, the carbon was conditioned by mixing in deionized water for ten minutes to allow for degassing and cleaning. The carbon was allowed to settle, and the suspended fines were decanted off with the supernatant. This conditioning was repeated until the supernatant was clear and colorless with no visible suspensions.

Detailed Description Text (59):
For column loading, the conditioned carbon was slurried and poured into a Plexiglas column having dimensions of about 1 inch diameter and 60 inches height. The final bed depth of the carbon was 36 inches. Deionized water was put through the column counter-currently to classify the carbon and remove any residual carbon dust.

Detailed Description Text (60):
Three samples were put through the carbon column, "A," "B," and "C," representing different manufacturing companies and separate facilities. One of the samples used during this test was "A" slurry previously concentrated using a Membralox Silverback.RTM. microfilter purification system available commercially from U.S. Filter Wastewater Group, Inc. in Warrendale, Pa. The concentrate was re-diluted with deionized water to simulate "as-received" characteristics.

Detailed Description Text (62):
The CMP slurry solutions containing hydrogen peroxide were passed through the carbon filter bed without prior removal of any of the alumina, silica particles in the CMP slurry solutions. During this stage of experimental, an influent pressure and a hydrogen peroxide content were monitored.

Detailed Description Text (68):
The results of Table 2 showed that carbon could remove hydrogen peroxide from CMP slurry solutions without entrapping the alumina, silica particles within the filter

bed.

Detailed Description Text (70):
Ion exchange tests were performed to remove copper from the alumina slurry without removing the slurry itself. Ion exchange tests were performed using iminodiacetic resin, a selective resin for heavy metal ion removal. The resins were conditioned prior to operation to be in the monosodium form as follows:

Detailed Description Text (71):
Backwash for 30 minutes to classify resin

Detailed Description Text (73):
Rinse with 20 bed volumes deionized water

Detailed Description Text (75):
Rinse with 20 bed volumes deionized water

Detailed Description Text (76):
Six (6) ion exchange tests were performed using the conditioning model listed above.

Detailed Description Text (78):
A first stage carbon column had the general characteristics: 1".times.24" glass column before the second stage of ion exchange resin, 18" of conditioned resin.

Detailed Description Text (79):
The carbon column effluent was piped directly to the ion exchange column.

Detailed Description Text (81):
The results showed an early break through of copper which was identified as caused by the monosodium conditioning of the resin.

Detailed Description Text (82):
After a substantial amount of carbon column effluent had been put through the ion exchange bed, regeneration was attempted. The column was first back washed with 2 liters of deionized water to remove residual solids from the resin bed. The expansion of the bed during backwash reached 80%. The regeneration cycle consisted of 485 ml of 4% sulfuric acid introduced in counter-current up-flow mode.

Detailed Description Text (84):
The resin "rocks up." Blue chunks form upon introduction of acid and do not break up easily.

Detailed Description Text (85):
Many fine white solids emerge from the resin which were not evident in the backwash.

Detailed Description Text (86):
Rinsing with 1 liter of deionized water did not loosen the "rocks."

Detailed Description Text (88):
An additive without slurry was tested to confirm that the resin would remove copper from the complexed solution without solids present.

Detailed Description Text (89):
The feed for the experimental was prepared by adding 20 ml of ammonium citrate/copper solution per gallon of deionized water.

Detailed Description Text (90):
General Characteristics: 1".times.24" glass column, 18" of conditioned resin.

Detailed Description Text (92):
A slurry with copper and no additive was tested to investigate if the resin could remove copper from a slurry solution without any additional completing agents.

Detailed Description Text (94):
General Characteristics: 0.6".times.24" glass column, 15" of resin.

Detailed Description Text (98):
General Characteristics: 0.6".times.24" glass column, 15" of conditioned resin.

Detailed Description Text (100):
The effectiveness of the same resin in hydrogen form was investigated on a pH-adjusted sample. The resin was conditioned as described below.

Detailed Description Text (101):
Backwash for 30 minutes to classify resin

Detailed Description Text (103):
Rinse with 20 bed volumes deionized water

Detailed Description Text (105):
General Characteristics: 0.6".times.24" glass column, 15" of resin conditioned into the Di Hydrogen Form.

Detailed Description Text (108):
As a result of an incomplete sample volume, the exhaustion run was terminated and 31.4 ml of the 54 ml of resin was removed for regeneration. The results are shown in FIG. 9 which depict an efficient regeneration of the 31.4 ml of resin with 10% HCl. Approximately 100% of the loaded copper was recovered.

Detailed Description Text (112):
A recycle test, diffusion head of ion exchange unit was conducted to investigate the ability of the slurry to pass through an ion exchange diffusing head without clogging over time.

Detailed Description Text (115):
A series of slurry agitation tests were performed to characterize the effect of slight, moderate, and severe agitation of the slurry samples. Filter pore sizes were 0.45 micron, 0.22 micron, and 0.10 micron. Results are shown in Table 9.

Detailed Description Text (118):
A column was partially loaded with copper from a CMP slurry. Because of an insufficient sample to allow for complete loading, a regeneration was conducted on the partially loaded column. The results of Example II had indicated that the use of sulfuric acid is only partially effective and can lead to clumping of the resin. Hydrochloric acid was used for procedure.

Detailed Description Text (119):
The resin from the top 7.0 inches of a partially loaded column was removed by siphon and loaded into another column. The following is the data for the "new" column used in the regeneration.

Detailed Description Text (120):
A 10% HCl solution was used for the regeneration of the ion exchange column. The test was run for 60 minutes at an average flow rate of 1.39 ml/min. A total volume of 83.4 ml or 2.65 Bed Volumes (BV) was processed through the column. The test was stopped after a low level of copper was detected. Data are shown in Tables 11 and 12, and a regeneration curve is shown in FIG. 9.

Detailed Description Text (121):
The regeneration process involved draining the "new" column of the deionized (DI) water contained within the column. The HCl solution was then pumped into the column up-flow at a rate of 3-4 ml per minute until the effluent reached the end of the tubing. At this point, the flow rate was adjusted to approximately 0.3 BV/ft.sup.3.

Detailed Description Text (124):
The process and apparatus of the present invention remove metal ions from wastewater by providing a carbon bed for receiving a wastewater feed containing metal ions in solution, wherein the wastewater feed contains solids sized in the range of about 0.01-1.0 .mu.m in an amount higher than about 50 mg/l, in combination with providing a ion exchange unit operation for receiving a carbon bed product stream from the carbon bed and for removing the metal ions from solution. The process and apparatus of the present invention remove metal ions from wastewater containing solids in an amount higher than about 100 mg/l, e.g., by way of example in an amount in the range of about 500-2000 mg/l.

Detailed Description Text (126):
In one aspect, the ion exchange unit operation includes means for contacting metal ions in the carbon bed product stream with a resin having a macroporous iminodiacetic functional group to attach the copper ions. In one aspect, the ion exchange unit operation includes means for contacting metal ions in the carbon bed product stream with a resin having a cross-linked polystyrene resin to attach the copper ions. In one embodiment, the cross-linked polystyrene resin has a bead size in the range of about 0.4 to 1.23 mm.

Detailed Description Text (127):
The process and apparatus of the present invention operate to remove metal ions from a wastewater from a byproduct polishing slurry. In one embodiment, the process and apparatus of the present invention operate to remove metal ions, e.g., such as copper metal ions, from a wastewater from a byproduct polishing slurry from the chemical mechanical polishing (CMP) of integrated circuits to attach the metal ions and form an environmentally clean water discharge product. By environmentally clean is meant a wastewater discharge stream to a municipal wastewater treatment plant such that the wastewater discharge stream contains copper ions in a concentration less than about 0.5 mg/l (500 ppm).

Detailed Description Paragraph Table (1):
Dissolved copper 5.0 mg/l Total suspended solids 1000.0 mg/l Oxidizing agents 300.0 mg/l Etchants 200.0 mg/l Complexing agents 400.0 mg/l DI water background 99%+ TDS 800 pH 6 to 7

Detailed Description Paragraph Table (2):
Bead size min. 90% 0.4-1.23 mm Effective size 0.55 (.+-.0.55) mm Uniformity coefficient 1.7 Bulk weight (.+-.5%) 800 g/l Density 1.18 g/ml Water retention 50-55 wt % pH range 0-14 Functional group iminodiacetic Structure macroporous Matrix crosslinked polystyrene Minimum Capacity 2.7 mg/l in H.sup.+ Form

Detailed Description Paragraph Table (12):
TABLE 10 pH vs. Soluble Copper Copper (mg/l) Chemical pH 0.45 micron filter Sulfuric acid 4 0.9 Sulfuric acid 3 5.2 Sulfuric acid 2.5 15.7 Sulfuric acid 2 17.3 Sulfuric acid 1.5 17.6 Sulfuric acid 1 17.6 Sulfuric acid/NaOH 3 17.3 Sulfuric acid/NaOH 4 13.8 Sulfuric acid/NaOH 5 6.8 Sulfuric acid/NaOH 9.5 0.79

Detailed Description Paragraph Table (16):
IX Resin Chelate Regeneration 10% HCl Bed Volume 31.36 mls. Bed Volume 0.00111 ft3 Total Copper Load- 1.264 #/ft3 (adjusted to account for the vol. of resin used for reg'n.) Total Copper Re- 1.271 #/ft3 Copper Recovered 100.6% Time Flow Rate Copper in Regen Total Copper (minutes) BV (ml/min.) (mg/l) Removed (#/ft3) 3 0.16 1.7 53

0.003 6 0.27 1.1 230 0.015 9 0.37 1.1 665 0.050 12 0.48 1.1 730 0.090 15 0.57 1.0 597 0.120 18 0.67 1.0 850 0.166 21 0.86 2.0 1,125 0.275 24 1.01 1.6 1,230 0.367 27 1.17 1.6 1,230 0.458 30 1.32 1.6 1,390 0.558 33 1.43 1.2 2,530 0.696 36 1.59 1.6 3,110 0.920 39 1.74 1.6 2,655 1.109 42 1.88 1.5 1,085 1.181 45 2.03 1.5 620 1.222 48 2.16 1.4 315 1.241 51 2.29 1.3 270 1.257 54 2.41 1.3 233 1.270 60 2.53 1.2 9 1.271

Other Reference Publication (2):
"Treatment and Water Recycling of Copper CMP Slurry Waste Streams to Achieve Environmental Compliance for Copper and Suspended Solids", Mary Reker et al., Semiconductor Fabtech--8.sup.th Edition, (8 pages total).

Other Reference Publication (4):
Rohm and Haas "Helpful Hints in Ion Exchange Technology", Robert Kunin, Sep. 1981, IE-73-63-74, (8 pages total).

Other Reference Publication (5):
"Industrial Wastewater Treatment by Granular Activated Carbon", Donald G. Hager,Industrial Water Engineering, Jan./Feb. 1974, pp. 14-28.

Other Reference Publication (6):
Lewatit.RTM.--"The Use of Ion Exchange for the Polishing of Water", F. Martinola et al., Bayer, Paper given at the VGB conference in Essen, Oct. 1985, (43 pages total).

CLAIMS:

1. A process for removing metal ions from wastewater, comprising:

(a) passing a wastewater feed containing hydrogen peroxide and metal ions in solution through a carbon bed, wherein said wastewater feed contains solids sized in the range of about 0.01-1.0 .mu.m in an amount higher than about 50 mg/l to form a carbon bed product stream; and

(b) passing said carbon bed product stream from said carbon bed to an ion exchange unit for removing said metal ions from solution.

7. The process for removing metal ions from wastewater as set forth in claim 5, wherein said step of passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises contacting metal ions in said carbon bed product stream with a resin having a macroporous iminodiacetic functional group.

8. The process for removing metal ions from wastewater as set forth in claim 5, wherein said step of passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises contacting said carbon bed product stream metal ions with cross-linked polystyrene resin to attach said copper ions.

9. The process for removing metal ions from wastewater as set forth in claim 8, wherein said step of passing said carbon bed product stream from said carbon bed to an ion exchange unit comprises contacting said carbon bed product stream metal ions with cross-linked polystyrene resin screened to provide a bead size in the range of about 0.4 to 1.23 mm with a tight uniformity coefficient of about 1.7 to attach said copper ions.

12. A process for removing copper ions in a byproduct polishing slurry wastewater from the chemical mechanical polishing of integrated circuits, comprising:

(a) providing a carbon bed for receiving a byproduct polishing slurry wastewater feed from the chemical mechanical polishing of integrated circuits, said byproduct polishing slurry wastewater feed containing copper ions in solution at a level in

the range of about 5-25 mg/l and hydrogen peroxide to reduce the concentration of said hydrogen peroxide and form a carbon bed effluent product stream having concentration levels of hydrogen peroxide less than about 0.1 mg/l, wherein said byproduct polishing slurry wastewater feed further contains solids sized in the range of about 0.01-1.0 .mu.m in an amount higher than about 500 mg/l;

(b) providing a ion exchange bed of cross-linked polystyrene resin having a bead size in the range of about 0.4 to 1.23 mm for receiving a carbon bed product stream from said carbon bed and further having a macroporous iminodiacetic functional group for removing said copper ions from solution;

(c) passing to said carbon bed a byproduct polishing slurry wastewater feed from the chemical mechanical polishing of integrated circuits, said wastewater feed containing copper ions in solution at a level in the range of about 5-25 mg/l, hydrogen peroxide, and solids sized in the range of about 0.01-1.0 .mu.m in an amount higher than about 500 mg/l and withdrawing a carbon bed product stream containing copper ions;

(d) contacting copper ions in said carbon bed product stream with said cross-linked polystyrene resin in said ion exchange bed to attach said copper ions and form an environmentally clean water discharge product; and

(e) regenerating said polystyrene resin with hydrochloric acid.

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L4: Entry 13 of 18 File: USPT Feb 14, 1989

DOCUMENT-IDENTIFIER: US 4804465 A
TITLE: Water treatment apparatus

Abstract Text (1):
A water treatment system including a pair of tank assemblies 110, 112 interconnected by a valve module 114 for controlling the communication of water to be treated to an on-line tank and the regeneration of an exhausted tank. Each tank assembly includes a compartment containing a water softening resin 125 and a compartment centering an iron filter media. The valve module is connected to a regeneration solution reservoir 150 by brine conduits 164, 220. A pumping assembly 192 automatically transfers a predetermined amount of a supplemental regenerant fluid from a well 168 into the regeneration solution reservoir at the conclusion of a regeneration cycle. The pumping assembly includes upper and lower chambers 194a, 196a communicating with the supplemental regenerant fluid reservoir 168 and brine supply conduits 164, 220 respectively. The lower chamber 196a contracts and expands in response to the depressurization and pressurization of the brine conduits and produces attendant expansion and contraction in the upper chamber 194a. The upper chamber draws a predetermined amount of regenerant fluid from the reservoir 168 and injects the fluid into the regeneration solution reservoir 150. Check valves 186, 202 control the direction of flow of the regenerant fluid from the regenerant well 168.

Brief Summary Text (2):
The present invention relates generally to water treatment and in particular to a new and improved water softening apparatus and method for treating water having a high iron content.

Brief Summary Text (4):
Water softeners of the "ion exchange" type typically include a resin tank through which hard water passes to exchange its "hard" ions of calcium and magnesium for the "soft" sodium ions from the resin bed. Regeneration of the resin bed is required periodically to remove the accumulation of hard ions and replenish the supply of soft ions. Regeneration is typically affected by flushing a brine solution through the resin bed. A water softener of this type is more fully described in U.S. Patent No. 3,891,552 issued June 24, 1975 to William Pryor and James W. Kewley, entitled CONTROL VALVE FOR WATER SOFTENERS, the disclosure of which is incorporated herein by reference.

Brief Summary Text (5):
Modern water softeners of the type disclosed in U.S. Patent No. 3,891,552 typically employ a brine tank which includes a reservoir and supply of salt disposed at a level above the bottom of the reservoir. A tube connected to a source of water establishes a path for water to flow to the reservoir. Upon the attainment of a predetermined level in the reservoir, the water reacts with the salt supply to produce a source of brine for regeneration of the resin bed. When regeneration is required, the brine is aspirated through the same tube that supplied water to the reservoir. The amount of water introduced to the brine tank after the regeneration cycle and the amount of brine aspirated from the tank during a regeneration cycle is controlled by a brine valve mechanism.

Brief Summary Text (6):
Commercially available water softeners generally include one or two tanks which contain the softening chemicals that form the resin beds. In a two tank water softener, one tank is regenerated and kept "off-line" while the other tank is "on-line". A control valve controls the communication of the tanks with the household water supply and controls the timing and sequence of regeneration. One such system and control valve is disclosed in U.S. Pat. No. 3,891,552. An improved control valve is described in U.S. Pat. No. 4,298,025. Both of these patents are owned by the present assignee and are herein incorporated by reference.

Brief Summary Text (7):
A prior art resin tank typically comprises an elongate cylinder in which the ion exchange resin is contained. A conduit, often called a riser pipe, extends downwardly from the top of the tank. A filter screen, mounted at the end of the conduit prevents the entry of resin into the conduit. An opening is formed in the top of the tank for discharging or admitting fluid depending on the direction of flow.

Brief Summary Text (8):
In many applications, the prior art water softeners such as the one described in the above referenced patent, performed satisfactorily. However, in some areas of the country "problem water" is encountered which is not easily treated by conventional water softening apparatus. In particular, in some regions, the water has a high iron content and low pH. When a typical water softening apparatus is used to treat this water the result if still considered unacceptable or only marginally acceptable. Although methods and apparatus are known for treating water having a high iron content and low pH, many if not most are considered uneconomical for home use or use a process that the average homeowner is not equipped to handle.

Brief Summary Text (10):
The present invention provides a new and improved water treatment apparatus which not only softens water, but is also capable of removing high levels of iron from the water and of adjusting the pH of the treated water to an acceptable neutral range of 6.8 -8.0.

Brief Summary Text (11):
In accordance with the invention, the water treatment system includes an ion exchange bed that is periodically regenerated, an iron filter for filtering the water to be treated prior to passage through the ion exchange bed, a reservoir for regeneration solution that is used to regenerate an exhausted ion exchange bed, and a control apparatus for determining the frequency at which the resin bed is regenerated. According to the invention, the iron filter is flushed during a regeneration cycle of the resin bed in order to flush out the accumulated iron.

Brief Summary Text (12):
In the preferred and illustrated embodiment, the resin bed and iron filter are contained in serially connected tanks. Preferably the water to be treated enters the tank containing the iron filter first and then exits and enters the tank containing the resin bed. After passing through the bed, the treated water leaves the ion exchange tank through an outlet connected to the household water supply.

Brief Summary Text (13):
The treating system also includes a source of regeneration solution that is passed through the ion exchange tank during the regeneration cycle in order to reactivate the ion exchange resin. In accordance with the invention, the regeneration solution is a composite regenerant. To achieve this feature, the system includes a conventional brine tank with a control valve. During a regeneration cycle, solution from the brine tank is conveyed to the resin bed being regenerated. In accordance with a feature of the invention, the system also includes a vessel containing a

supplemental regenerant fluid and a metering arrangement for periodically adding to the regenerant to the brine solution to create a composite regeneration solution.

Brief Summary Text (14):
In the preferred embodiment, the metering arrangement responds to the termination of brine solution flow during the regeneration cycle and adds a predetermined quantity of a supplemental regenerant fluid to the brine tank. In the illustrated embodiment, the control apparatus for controlling the water treatment system preferably comprises a valve of a type described in U.S. Pat. Nos. 3,891,552 and/or 4,298,025. This type of control valve includes a venturi arrangement that is activated during the regeneration cycle and aspirates regeneration solution into the tank being regenerated. In particular, the brine conduit is connected to the throat of the venturi. During the regeneration cycle, water is communicated to the venturi which draws regeneration solution from the brine conduit as it passes through the venturi throat. The water and regeneration solution is then conveyed to the tank being regenerated. Regeneration solution travels from the brine tank to the venturi as long as water continues to flow through the venturi and as long as the brine control valve remains open. When a predetermined amount of brine solution has left the brine tank, the brine control valve closes and prevents further brine solution from entering the control valve.

Brief Summary Text (16):
The addition of the supplemental regenerant fluid to the brine reservoir occurs automatically at the conclusion of each regeneration cycle and is accomplished without operator intervention. The resulting composite regeneration solution enhances the regeneration of the resin bed without significantly adding cost to the overall system.

Brief Summary Text (17):
In accordance with another feature of the invention, the tanks containing the resin bed and iron filter are interconnected directly and stacked vertically. In accordance with this feature, the resin tank includes an opening at each end. The tank containing the iron filter, however, includes only a single opening that is coupled to one end of the ion exchange tank. Once connected, the tanks are axially aligned thus enabling a single riser tube to extend from the top of the ion exchange tank and into the iron filter tank. The opening of the riser tube is located near the base of the iron filter tank and forms an inlet to the tank structure such that water to be treated communicated to the riser tube enters the overall tank structure at or near the bottom of the iron filter. From there is travels upwardly through the iron filter, is discharged from the top of the iron filter tank and immediately enters the bottom of the ion exchange tank. The water then travels through the ion exchange bed and is discharged to an outlet at the top of the ion exchange tank.

Brief Summary Text (18):
The iron filter preferably comprise a floating media such as "hollow macrospheres" sold by the 3M Company. By using a floating filter media, during normal operation, the media tends to rise towards the top of the iron filter tank. The iron filtered from the water then tends to accumulate at the bottom of the filter media. In the preferred operation, downflow regeneration is employed so that regeneration solution and backwash water enters the top of the resin tank and exits the bottom of the iron filter tank. During the backwash process the iron accumulated by the iron filter is easily flushed out of the iron filter tank because the accumulated iron is simply pushed downwardly from the bottom of the filter media and travels out the riser tube.

Brief Summary Text (19):
It should be apparent that a new and improved water treatment system is disclosed. The system not only softens hard water but also removes iron from "problem water" and adjusts its pH to a neutral range. The iron filter for removing the iron from

the water being treated, prior to entering the ion exchange bed, is flushed automatically during a regeneration cycle to remove the accumulated iron.

Drawing Description Text (2):
FIG. 1 is a schematic representation of a water softening system embodying the present invention;

Detailed Description Text (2):
FIG. 1 schematically illustrates a water treatment system constructed in accordance with the preferred embodiment of the invention. The system includes a pair of resin tank assemblies 10, 12 interconnected by a control valve module 14. The control valve module 14 can take many forms. In the preferred embodiment, the control valve module is the type described in U.S. Pat. Nos. 4,298,025 and 3,819,552.

Detailed Description Text (3):
As fully described in the above referenced patents, the control assembly 14 controls the combination of a source of water to be treated, indicated generally by the reference character 16 with the treatment tank assemblies 10, 12; the communication of the tanks with an outlet indicated by the reference character 18; and, the regeneration of an exhausted tank assembly. In FIG. 1 the fluid flow paths are illustrated for a condition in which the tank assembly 10 is "on-line" and the tank assembly 12 is being regenerated. As fully described in the above referenced patents, the control valve 14 also includes passages, valves, etc. (not shown) for placing the tank assembly 12 "on-line" and for regenerating the tank assembly 10. As seen in FIG. 1, the on-line tank assembly 10 receives water from the inlet 16. The water to be treated flows down a riser pipe 20 and enters the tank assembly near the bottom. From there the water moves upwardly traveling through the resin (not shown) in the tank assembly, exiting near the top and into a tank outlet passage 22 connected to the outlet 18. It should be understood that the control module 14 includes valving for controlling the communication of the inlet 16 with the inlet pipe 20 and the communication of the tank outlet passage 22 with the outlet 18. To simplify the explanation, the fluid circuits and valves have been omitted.

Detailed Description Text (4):
The tank assembly 12 is regenerated by passing a regeneration solution, in a counterflow direction, through the resin. In many if not most conventional, residential softening systems, a brine reservoir 30 forms a source of regeneration solution. During regeneration, a valve 32, indicated schematically, opens to communicate softened water in the outlet passage 22 with a venturi 34. The opposite end of the venturi 34 is connected to the outlet 22' of the tank being regenerated, in this case the tank assembly 12. The throat 34a of the venturi 34 is connected to a brine conduit 36 that extends into fluid communication with a brine well 38 located in the brine reservoir 30. The action of water passing through the venturi 34 draws or aspirates brine solution from the brine reservoir. The brine solution is entrained in, and diluted by, the softened water and flows through the tank assembly 12. Near the bottom of the tank assembly 12, the regeneration solution enters the inlet pipe 20' and is dumped to a drain, indicated schematically by the reference character 42. The inlet pipe 20' is connected to the drain 42 by the drain valve 44, indicated schematically. The amount of brine communicated to the control module 14 is controlled by a brine valve 46 (indicated schematically). An example of a brine valve is disclosed in U.S. Pat. No. 4,336,134, which is owned by the reference assignee, and is herein incorporated by reference.

Detailed Description Text (5):
After the flow of brine solution is terminated, softened water continues to flow through the tank assembly 12 to "backwash" the resin or in other words to rinse the brine solution from the regenerated resin.

Detailed Description Text (6):

At the end of a regeneration cycle, the flow of wash water to the tank assembly being regenerated (in this case tank assembly 12) is terminated and the brine reservoir is refilled with water, usually under the control of the brine valve, to replenish the regeneration solution used during the previous regeneration cycle. The replenishment is usually controlled by the brine valve 46. With the illustrated and preferred control module 14, the replenishment of the brine reservoir 30 is achieved through the brine conduit 36. It should be apparent that if the drain valve 44 is closed to prevent discharge of fluid to the drain 42, the flow of softened water through the venturi will terminate; if the brine valve 46 is opened, the flow of softened water will proceed from the throat 34a of the venturi to the brine reservoir 30 by way of the brine conduit 36.

Detailed Description Text (7):
In accordance with the preferred embodiment, each tank assembly 10, 12 includes a water softening resin bed and an iron filter bed 48, 50 respectively. In the preferred embodiment, the beds are housed in a pair of serially connected tanks 10a, 10b and 12a, 12b respectively.

Detailed Description Text (11):
In the preferred method, the pressurization and depressurization of the brine conduit 36 during the regeneration cycle operates the pump 60. During the "brining" portion of the regeneration cycle, brine solution flows from the brine well 38 to the venturi 34. When sufficient brine has been dispensed, the brine valve 46 closes to terminate the flow of regeneration solution. The termination of brine flow produces a suction pressure in the brine conduit 36 which acts on the diaphragm 62 and contracts the upper chamber 64. Upward movement of the diaphragm 62 in turn causes expansion of the lower chamber 66 and produces a suction pressure in the regenerant supply line 54. The check valve 58 prevents flow out of the brine reservoir 30; the check valve 58 opens to allow the flow of supplemental regenerant fluid from the regenerant reservoir 52 into the chamber 66. As discussed above, at the conclusion of the regeneration cycle, the brine conduit 36 becomes pressurized as water flows through the line to refill the brine reservoir 30 (via the throat 34a of the venturi 34). The pressurization of the brine conduit 36 expands the chamber 64 thus forcing the stored regenerant fluid from the lower chamber 66. Since the check valve 56 is oriented to prevent flow from the conduits 70, 54 into the regenerant reservoir 52, the expelled regenerant fluid is discharged into the brine reservoir 30. It should be apparent, that a pre-determined quantity of the supplemental regenerant fluid is automatically injected into the brine reservoir 30, at the conclusion of each regeneration cycle thus creating a composite regeneration solution in the reservoir.

Detailed Description Text (12):
Turning now to FIGS. 2 and 3, the construction of a water treatment system embodying the present invention is illustrated. Referring in particular to FIG. 2, the system includes a pair of tank assemblies 110, 112 interconnected by a control valve assembly 114. The control valve assembly 114 includes an inlet 116 for water to be treated and an outlet 118 for discharging treated water into the household water supply (not shown). The tank assembly 110 is interconnected with the tank 112 by a fitting 119 and a pair of parallel conduits 121a, 121b. As fully described in U.S. Pat. Nos. 3,891,552 and 4,298,025, the control valve assembly 114 controls the regeneration of the tank assemblies 110, 112 and determines which of the tank assemblies is "on line".

Detailed Description Text (14):
In the preferred and illustrated construction, the top tank houses a water softening resin 125 whereas the lower tank houses an iron filter media 127. For purposes of explanation the resin 125 and filter media 127 are shown only in the tank assembly 112. It should be understood that the tank assembly 110 also includes the resin and filter media.

Detailed Description Text (15):
In the preferred embodiment, when a tank is "on line", service is "up flow". In particular, water to be treated enters near the bottom of the lower tank, e.g., tank 110b, travels upwardly through both tanks and exits at the top of the upper tank, e.g., tank 110a. In accordance with the preferred construction, a riser tube 130 extends downwardly through both tanks 110a, 110b and opens at the bottom of the tank 110b. The tank assembly 112 includes a similar riser tube. The fitting 119 includes passages for communicating the conduit 121a with the riser pipe 130. A filtering screen 132 is mounted at the end of the riser pipe to prevent material/resin in the tank from entering the riser pipe 130.

Detailed Description Text (16):
Fluid to be treated enters a tank assembly at the bottom of the lower tank. When the tank assembly 110 is active or "on-line", the water to be treated exits the riser pipe 130 through the filtering screen 132 and flows upwardly through the iron filter material 127. The connector 123 which couples the tanks 110a, 110b together, also includes upper and lower filtering screens 134, 136 and passages (not shown) for placing the tanks in serial, fluid communication. The water leaves the tank 110b through the coupling 123 and enters the upper tank 110a, which in the preferred embodiment, contains the water softening resin 125. The water then continues its upward travel through the upper tank 110a and leaves the upper tank through a filtering screen 138a mounted to the fitting 119. the screens 134, 136 and 138 prevent the filter material 127 and resin 125 from intermixing and/or from leaving the tanks.

Detailed Description Text (17):
In the preferred embodiment, the control valve 114 which is more fully described in Pat. No. 4,298,025 causes the regeneration of an exhausted tank assembly to occur in a downflow direction. Regeneration solution enters at the top of the upper tank (e.g. tank 110a), travels downwardly through the upper and lower tanks 110a, 110b and exits by way of the riser tube 130. By counterflowing the regeneration fluid, the regeneration of the water softening resin 125 is enhanced and, more importantly, iron accumulated by the iron filter material 127 in the lower tank 110b is more easily and more completely flushed from the filtering bed in tank 110b.

Detailed Description Text (18):
Turning now to FIG. 3, the regeneration solution reservoir (element 30 in FIG. 1) includes an open top tank 150 and a complemental tank lid 151. A screen or perforate plate 152 is supported above the bottom of the reservoir 150 by a support 154. As is known in the art, the perforate plate 152 supports a supply of salt (not shown) above the bottom of the reservoir. Water is added to the reservoir until a portion of the salt supply is submerged. The submerged salt then dissolves to form a brine solution. A fitting 156 is mounted in the side of the reservoir and forms an overflow to prevent an excess water level from developing in the reservoir.

Detailed Description Text (19):
A brine or regeneration solution well 160, formed by a tubular member mounted vertically along the wall of the reservoir, extends through the perforate plate and opens at or near the bottom of the reservoir. A brine valve 162 which may be the form shown in U.S. Pat. No. 4,336,134 is positioned a the lower end of the brine well and controls the flow of regeneration solution out of the reservoir 150 and the inflow of replacement water into the reservoir. The brine valve 162 is connected via a conduit 164 to the control valve apparatus 114. (A schematic representation of the connection is shown in FIG. 1 and denoted as conduit 36). As explained above and shown in FIG. 1, the conduit 164 is connected to a venturi mechanism in the control valve 114 which becomes operative to cause the aspiration of regeneration solution during a regeneration cycle.

Detailed Description Text (21):

In the preferred embodiment, a predetermined amount of the supplemental regenerant fluid is drawn from the regenerant well 168 during a regeneration cycle and is injected into the brine or regeneration solution at the conclusion of the regeneration cycle. The apparatus for achieving this feature includes an inlet assembly 180 which includes a filtering screen 182 mounted below a set of radially extending arms 184 which serve to locate the filter near the center of the regenerant well 168. The assembly 180 also includes a ball check valve 186 and associated fitting 188. The assembly is disposed at or near the bottom of the regeneration well.

Detailed Description Text (26):
As discussed above, during a regeneration cycle, as for example when the tank assembly 112 is being regenerated, softened water is communicated (via valve 32 shown in FIG. 1) to a venturi 34 (also shown in FIG. 1). The flow of water through the venturi draws regeneration solution from the brine well through the conduits 164, 220 (conduit 36 in FIG. 1). When a predetermined amount of the regeneration solution is drawn from the reservoir 150, the brine valve 162 closes. The continued flow of softened water through the venturi 34 develops a suction pressure which causes the diaphragm 198 to move downwardly (as viewed in FIG. 3) and contract the lower chamber 196a. The attendant expansion of the upper chamber 194a causes regeneration fluid in the regeneration well 168 to be drawn into the upper chamber 194a through the conduit 190 and check valve 186. The amount of solution drawn corresponds to the change in volume of the upper chamber 194a. The check valve 202 prevents air from being drawn into the conduit 204.

Detailed Description Text (27):
At the conclusion of the regeneration cycle, the flow of softened water through the tank assembly 112 is terminated by closing the drain valve 44 (shown in FIG. 1). When the flow path through the tank is interrupted, the venturi 34 and hence the brine conduits 220, 164 (conduit 36 in FIG. 1) are pressurized and a replenishment of the brine reservoir 150 begins. The pressurization of the brine conduit exerts an expansion force in the lower chamber 196a and causes the diaphragm to expand upwardly, expanding the volume of the lower chamber while concurrently decreasing the volume of the upper chamber. This causes at least a portion of the regenerant fluid stored in the upper chamber 194a to be expelled into the conduit 204. Since the check valve 186 prevents return flow of the regenerant fluid into the regeneration well 168, the check valve 202 opens to allow the expelled fluid to be injected into the tube 208. From there it enters and mixes with the brine solution to form a composite regeneration solution in the reservoir 150.

Detailed Description Text (28):
It should be apparent that the introduction of the supplementary regeneration fluid occurs automatically at the conclusion of each regeneration cycle and is achieved without the use of externally powered controls. The apparatus, being relatively uncomplicated, enables a homeowner to treat problem water which in the past was precluded by the high cost of equipment.

Detailed Description Text (29):
The disclosed apparatus when used with appropriate iron filter material and water softening resins can effectively remove both ferric and ferrous iron from the water supply and in addition adjust a low pH upwardly to a neutral range of 6.8-8.0.

Detailed Description Text (30):
Various water softening resins and iron filter materials can be utilized in the system. As an example, for a low pH water supply having high amounts of undissolved and dissolved iron, the ion exchange resin may be an acrylic based, weak acid carboxylic resin. A resin of this type is sold by the Rhom and Haas Company under the trademark AMBERLITE. For regeneration, in addition the standard brine solution, a supplemental regenerant is required. The composition of a supplemental regenerant that performs satisfactorily is disclosed in U.S. Pat. No. 4,116,860. Supplemental

regeneration compositions other than that described in the patent are also possible and contemplated by the present invention. It should be understood that the quantity of the regenerant fluid added to the brine reservoir at the conclusion of each regeneration cycle can be adjusted by selectively sizing the pump chambers 194, 196 and/or alternately by adjusting the concentration of the supplemental regenerant fluid in the regeneration well 138.

Detailed Description Text (31):
Various materials can also serve as the iron filter media in a lower tank. An example of such a material that is found to produce satisfactory results is known as 3M brand hollow macrospheres sold by the 3M Company. It is believed that other materials can serve as n iron filter media.

Detailed Description Text (32):
In the preferred embodiment, a floating filter media is used in the lower tank. As described earlier, when a tank assembly is on-line, service is "upflow". Regeneration is conducted in a counterflow or downflow direction. Referring to FIG. 2, water to be treated enters the lower tank 110b by way of the riser tube 132, The water travels upwardly through the filter tank 110b and then enters the resin tank 110a. During operation, the floating filter media rises to the top of the lower tank 110b and traps dissolved iron and other solids carried by the water and prevents the material from entering the upper tank. When the tank assembly 110 is regenerated, the regeneration solution and wash water enters at the top of the upper tank 110a and travels downwardly through both tanks 110a and 110b, leaving the lower tank 110b through the riser tube. This downward flow of regeneration solution and wash water flushes the accumulated, undissolved iron (and other solids) from the floating filter media and carries it out of the tank and discharges it to a drain.

Detailed Description Text (33):
The use of a floating filter media thus enhances the filtering capability of the system. When the tank is on line, filter media congregates at the top of the tank to inhibit the undissolved iron from entering the resin tank 110a. During down flow regeneration and wash, the accumulated iron is easily dislodged from the media and flushed from the system.

CLAIMS:

1. For a water softening apparatus having an ion exchange bed, a reservoir of brine solution for regenerating the bed and an aspirator for drawing solution from said reservoir and delivering it to the bed during a regeneration cycle, a metering apparatus for adding a supplemental regenerant fluid to the solution reservoir to produce a composite regeneration solution, comprising:

(a) a housing and an expansible member in said housing dividing said housing into two expansible, non-communicating chambers, one of said chambers communicating with said aspirator such that when said aspirator becomes effective, said one expansible chamber contracts;

(b) said other expansible chamber communicating with a vessel containing supplemental regenerant fluid and operative to expand as said one chamber contracts to thereby draw a quantity of said supplemental regenerant fluid from said vessel;

(c) means for pressurizing a conduit communicating with said aspirator at the conclusion of said regeneration cycle such that said one chamber is caused to expand thereby expelling said supplemental regenerant fluid from said other chamber and into a conduit communicating with said solution reservoir.

3. A water treatment system, comprising:

(a) a tank assembly including an upper tank containing a water treatment resin and a lower tank containing a floating filter media;

(b) means serially connecting said upper and lower tanks;

(c) structure extending through said upper and lower tanks defining an inlet for water to be treated near the bottom of the lower tank, said structure communicating with a source of water to be treated located near the top of the upper tank;

(d) a reservoir of brine solution for regenerating the water treatment resin in the upper tank;

(e) an aspirator for drawing solution from said reservoir and delivering it to said upper tank during a regeneration cycle; and,

(f) a metering apparatus for adding a supplemental regenerant fluid to the solution reservoir to produce a composite regeneration solution, comprising:

(i) a housing and an expansible member in said housing dividing said housing into two expansible, non-communicating chambers, one of said chambers communicating with said aspirator such that when said aspirator becomes effective, said one expansible chamber contracts;

(ii) said other expansible chamber communicating with a vessel containing supplemental regenerant fluid and operative to expand as said one chamber contracts to thereby draw a quantity of said supplemental regenerant fluid from said vessel;

(iii) means for pressurizing a conduit communicating with said aspirator at the conclusion of said regeneration cycle such that said one chamber is caused to expand thereby expelling said supplemental regenerant fluid from said other chamber and into a conduit communicating with said solution reservoir.

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L5	L4 and reuse	12	L5
L4	L3 and ion exchange	65	L4
L3	L2 and salt	70	L3
L2	regenerant same membrane	88	L2
L1	regenerantsame membrane	0	L1

END OF SEARCH HISTORY

L2: Entry 6 of 14 File: USPT Aug 3, 1999

DOCUMENT-IDENTIFIER: US 5932099 A

**** See image for Certificate of Correction ****

TITLE: Installation for biological water treatment for the production of drinkable water

Brief Summary Text (26):
The means used to add a pulverulent material to the reactor may therefore be used to add to the water present in the reactor any non-reactive pulverulent material serving as a support for the biomass used, such as for example sand or anthracite, and also to add to said water one or more reactive powders of various types. It will be noted that for the present description the terms "reactive powders" shall be understood to mean any powder likely to react with one of the compounds in the drinking water to be treated, either by adsorption or by ion exchange, with a view to removing the content of these compounds in said water to be treated. These powders must also have physical characteristics of hardness, abrasiveness and density, allowing their fluidization in the water present in the reactor and making them compatible with the membranes used.

Detailed Description Text (5):
An ultrafiltration module 11 consisting of outer pressure hollow fibres (filtration from outside to inside the membrane) representing a filtering surface of 12 m.sup.2 is immersed in reactor 7. The compactness and flux of these immersed membranes 11 allow treatment rates to be obtained that are comparable to existing sand filters, that is to say in the region of 6 to 8 m.sup.3 /m.sup.2 /h. The permeate exiting this module is evacuated by pipe 12 after aspiration pumping by pump 12a. The treated water (TW) is collected at the exit of pipe 12 and passes into tank 13 before leaving the plant.

Detailed Description Text (6):
In accordance with the invention, means of adding pulverulent materials 10, consisting of a bubble mixer, can be used to add to the water transiting through reactor 7 both non-reactive powders, such as sand or anthracite serving as a fixation support for a biomass, and reactive powders in continuous or intermittent manner. These reactive powders are preferably made up of powdered activated carbon (PAC), zeolites, clays or ion exchange resins and are added to the water to be treated in relation to the load of water to be treated and to the temperature thereof.

Detailed Description Text (9):
With said aeration means 9 it is possible to maintain in suspension the powders present in reactor, to mix the water with these powders, to provide bacteria with necessary oxygen and to shake the immersed membranes to prevent fouling.

CLAIMS:

2. Plant according to claim 1, characterized in that said reactive powder is chosen from the group made up of powdered activated carbon, zeolites, ion exchange resins and clays.

L8: Entry 4 of 14 File: USPT Oct 15, 2002

DOCUMENT-IDENTIFIER: US 6464881 B2
TITLE: Inorganic nanofiltration membrane and its application in the sugar industry

Brief Summary Text (14):
The use of this membrane thus makes possible efficient recycling of the saline effluent resulting from the regeneration of the ion-exchange resins and thus a significant reduction in the amounts of sodium chloride and of water necessary for the manufacture of the solutions for the regeneration of the resins.

Detailed Description Text (2):
Use is made, for the treatment of a saline effluent resulting from the regeneration of the ion-exchange resins employed in a process for refining cane sugar, of a module (of approximately 1 m.sup.2) containing 9 inorganic nanofiltration membranes according to the invention; the nanofiltration membrane layer of each of these 9 membranes is a zirconia layer obtained by a process of sol-gel type in accordance with the preferred embodiment of the invention as described above (drying temperature: 80.degree. C.; heat treatment temperature: 400.degree. C.), deposited on a Kerasep.RTM. ultrafiltration membrane having a cutoff threshold of 15 kD, the support (Al.sub.2 O.sub.3 /TiO.sub.2 monolith, with a TiO.sub.2 /(Al.sub.2 O.sub.3 +TiO.sub.2) ratio by weight of 25%,) having a diameter of 20 mm and a length of 856 mm and containing 19 channels; in each of the 9 membranes, the microfiltration membrane layer is made up of titanium oxide and the ultrafiltration membrane layer is made up of zirconia.

Current US Cross Reference Classification (3):
210/651

CLAIMS:

10. A process for the treatment of saline effluents resulting from the regeneration of the ion-exchange resins employed in the refining of cane sugar comprising the step of filtering said effluents with a membrane as defined in claim 1.

L1: Entry 1 of 49 File: USPT Aug 17, 2004

DOCUMENT-IDENTIFIER: US 6776913 B1
TITLE: Water softening method and apparatus for use therewith

JANGBARWALA

Brief Summary Text (6):
A common method of softening water, such as in small domestic water softeners, involves the process of ion exchange. Ion exchange is a process whereby a water solution is passed through a column of a material that replaces one kind of ion in solution with another kind. Such materials are known as ion exchange resins. Home and commercial water softeners generally contain cation-exchange resins. These resins consist of insoluble macromolecular substances to which negatively charged groups are chemically bonded. The negative charges are counterbalanced by ions such as sodium ions. When hard water containing the calcium or magnesium ion passes through a column of this resin, the sodium ions in the resin are replaced by calcium or magnesium ions. The reaction may be generalized as follows for calcium:

Brief Summary Text (8):
Water that has passed through the column containing the ion exchange resin contains sodium ions in place of calcium or magnesium ions, and has been softened. Once the resin has been completely converted to a calcium and/or magnesium salt, it can be regenerated by flushing the column with a concentrated solution of sodium chloride to reverse the previous reaction.

Brief Summary Text (10):
Once the resin has been completely converted to the calcium or magnesium salt, the resin must be regenerated. During regeneration, most softeners flow brine (which is formed by dissolving common rock salt in water) in the same direction as the service flow, and direct the water from the riser tube through the multi-port valve to a common drain, which is generally connected to a sewer. Some softeners may use a countercurrent flow of brine, but also direct all waste to the drain.

Brief Summary Text (11):
The regeneration process generally includes several steps, including a backwash, brine injection, a slow rinse and a fast rinse. While there may be some slight variations in different water softeners (for example, the sequence of the steps or the direction of flow may be different for some configurations), most water softeners generally utilize the same regeneration principles.

Brief Summary Text (13):
The step of brine injection generally involves opening an inlet valve to an eductor/injector. The eductor/injector is generally a venturi valve. The inlet valve is connected to a brine tank, such as with a flexible tube. Brine in the brine tank is formed by water and rock salt that a user puts in the brine tank periodically. Water is generally provided by a step in the regeneration process which directs water through the multi-port valve to the brine tank. The brine tank generally does not require any agitation, rather it simply saturates by soaking in the salt. The brine injection step includes sending city water at full pressure past the venturi valve, thereby causing a pressure gradient and sucking brine in from the injector to mix with the city water (or water from other water sources, such as well water) used to cause the pressure drop. This mixture is directed through the resin bed, up the riser tube, and out the common drain. The cycle is

timed to allow the resin exposure to a specific mass of sodium chloride, which is directly proportional to the capacity desired. Generally, the maximum salt required for achieving maximum resin regeneration capacity is exposed to the resin. After a specific amount of time has elapsed, therefore, the brine inlet valve is closed.

Brief Summary Text (14):
During the slow rinse step, city water (or water from a given water source) continues to be sent through the venturi valve. The venturi valve now acts as a flow control device and sends a slow stream of water to the resin bed, thereby rinsing the salt out. The waste is directed to the city drain. During the fast rinse step, city water is allowed to flow at full flow through the resin bed and the water is then directed to the city drain. This step packs the resin bed as well as purges any remaining salt out of the resin vessel. During this cycle, most water softeners also open the brine valve and refill the brine tank. A miniature float check valve in the brine tank shuts off flow when the brine tank has reached its capacity.

Brief Summary Text (17):
Accordingly, the water softener industry has been aggressively attempting to improve the efficiency of the devices they manufacturer. Devices currently on the market, however, do not reduce the volume of regeneration water enough to economically eliminate discharge to the sewer. Accordingly, it can be seen that there is a need for a new method for softening water with ion exchange which achieves a drastic reduction in waste volume. Further, it can be seen that there remains a need for a new water softener that reduces the waste volume sent to municipality waste treatment systems, and which allows the salt waste to be economically disposed of through alternative disposal routes. The present invention is directed to meeting these needs.

Brief Summary Text (24):
Accordingly, the present invention provides a water softening apparatus that is adapted to be placed in fluid communication with a water drain, a processing device, a water source that provides water containing undesired ions, and a water tap that dispenses water for consumption, wherein the water softening apparatus is operative to remove the undesired ions from water processed thereby. The water softening apparatus comprises a resin vessel containing an ion-exchange resin that is capable of chemically shifting between an active state and an exhausted state, a regenerant reservoir adapted to receive a regenerant solution containing selected preferred ions, and a manifold in fluid communication with the resin vessel and the regenerant reservoir.

Brief Summary Text (27):
The manifold is preferably a modified Autotrol Series 169 multi-port valve. The ion-exchange resin may be a shallow shell/shortened diffusion path resin or small bead size resin, and is preferably a Purolite SST or Purolite C100FM resin.

Brief Summary Text (29):
The present invention is further directed to a method for softening water that contains undesired ions. The method comprises providing an ion-exchange resin, contacting the ion-exchange resin with the water that contains the undesired ions when the ion-exchange resin is shifted toward its active state, contacting the ion-exchange resin with a regenerant solution containing the selected preferred ions when the ion-exchange resin is shifted toward its exhausted state so as to form a waste solution containing the undesired ions, and collecting the waste solution thereby to permit selective disposal of the undesired ions via a processing device that is separate from a drainage line.

Brief Summary Text (30):
The ion-exchange resin may be provided in a resin vessel that is sized and adapted to receive a selected volume of a fluid. The step of contacting the ion-exchange

resin with the regenerant solution may include first contacting the ion-exchange resin with the selected volume of the regenerant solution thereby to displace the selected volume of water from the resin vessel, and passing the selected volume of water to a water drain. The step of contacting the ion-exchange resin with the regenerant solution may include transporting, such as by pumping, the regenerant solution from a regenerant reservoir into the resin vessel, and may include contacting the ion-exchange resin with between 0.25 and 2.0 bed volumes of the regenerant solution. The step of collecting the waste solution may include transporting the waste solution to an evaporation device.

Brief Summary Text (31):
The method may further include the step of rinsing the ion-exchange resin with water thereby to form a rinse solution and thereafter transporting the rinse solution to the regenerant reservoir, and the step of adding rock salt to the regenerant reservoir, thereby to form a brine solution from the rinse solution.

Drawing Description Text (5):
FIG. 3(a) is a diagrammatic view of the brine draw cycle step according to the method of the present invention;

Drawing Description Text (6):
FIG. 3(b) is a diagrammatic view of the apparatus according to the present invention showing the direction of fluid flow during the brine draw cycle;

Drawing Description Text (9):
FIG. 5(a) is a diagrammatic view showing the rinse/brine refill cycle step according to the method of the present invention;

Drawing Description Text (10):
FIG. 5(b) is a diagrammatic view of the apparatus according to the present invention showing the direction of fluid flow during the rinse/brine refill cycle;

Detailed Description Text (2):
The present invention is directed to a method for softening water which limits or reduces the amount of waste sent to municipality water treatment systems, preferably to eliminate any high dissolved solids-bearing waste to such treatment systems. The present invention also is directed to an apparatus for softening water for use with the method of the present invention. In addition, the present invention is directed to a system for softening water for use with the existing water and drainage lines of a home residence or industrial complex. In particular, the present invention utilizes ion exchange resins with very fast kinetics. In addition, the present invention utilizes the flow of specific volumes of solution, which are relative to the bed volume, or liquid equivalent of the volume of resin in the softener. This volume allows the resin to be exposed to a specific mass of sodium chloride which is directly proportional to the maximum rated capacity of the resin. The present invention allows the salt bearing waste to be separated from the non-salt waste, thereby to allow the salt bearing waste to be separately disposed. In addition, the present invention allows the water used to rinse the resin to be additionally used for the formation of brine for use in the next cycle.

Detailed Description Text (3):
The method for softening water includes a service cycle, a brine draw cycle, a waste draw cycle and a rinse/brine refill cycle. The service cycle includes passing city water from an inlet into a resin vessel where ion exchange occurs, and thereafter passing the softened water through an outlet and to the desired water supply. The brine draw cycle includes pumping a specific volume of brine from a brine tank into the resin vessel, thereby to push the clean water that was in the resin vessel through a first drain, which may go to the municipality waste treatment system. The waste draw cycle includes continuing to pump the brine into the vessel thereby to push the solution containing the regeneration waste solution

from the ion exchange resin, which contains the contamination and salt that is objectionable to the municipalities, through a second drain that is not directed to the city municipality waste treatment system. The rinse/brine refill cycle sends a specific volume of city water, equal to the volume of brine used, through the inlet, through the resin vessel, and to the brine tank, thereby to rinse the resin vessel as well as refill the brine tank.

Detailed Description Text (6):
It is preferred that resins with very fast kinetics be utilized, such as those which have faster kinetics due to very small bead size or because the ion exchange region is only on the surface of the bead.

Detailed Description Text (7):
The present invention includes a water softening apparatus for use with the method of the present invention. The apparatus includes a resin vessel; a manifold, or multi-port valve; and a regenerant reservoir, or brine tank. The manifold is in fluid communication with an inlet and three outlets. The manifold includes various fluid pathways between the inlet, outlets, the resin vessel and the regenerant reservoir. Valves disposed in the fluid pathways permit configuration of various valve states that allow fluid flow according to the various water softening, regeneration and rinse cycles. The water softening apparatus according to the present invention may further include a pump disposed in fluid communication with the regenerant reservoir and the manifold.

Detailed Description Text (9):
With reference to FIG. 1, it may be seen that the method of the present invention includes a service cycle 12, a brine draw cycle 14, a waste draw cycle 16 and a rinse/brine refill cycle 18. In addition, the present invention may include a backwash cycle 20. With reference to service cycle 12, it may be seen that city water 50 from a water source is moved from inlet 22 to resin vessel 24 where the hard city water 50 from inlet 22 is softened by ion exchange. It should be understood that water from other sources, such as well water or water internal to an industrial plant, may be used instead of city water, and references herein to "city water" should be understood to encompass water from any selected water source. The resulting soft water 52 is then moved from resin vessel 24 to a first outlet 26 which is in fluid communication with the home or industrial complex water tap. The brine draw cycle 14 includes moving regenerant solution 54, such as brine solution, from the regenerant reservoir 28, or brine tank, to the resin vessel 24. The brine solution contacts an ion exchange resin in the resin vessel and exchanges sodium ions for the metal ions removed from the hard water during the softening process, thereby regenerating the ion exchange resin. It should be understood that appropriate regenerant solutions may be substituted for the brine solution for a given ion-exchange resin. Clean water 56, which contains no or little regenerant wastes, is pushed out of the volume of the resin vessel while brine 54 is being pushed into the resin vessel. This clean water 56 is sent to the second outlet 30, or first drain, whereby the clean water 56 is allowed to be sent to the municipality waste treatment system or other disposal means.

Detailed Description Text (10):
As brine 54 continues to be pushed into resin vessel 24, the waste draw cycle 16 removes the waste water solution 58 resulting from the regeneration of the resin in the resin vessel 24 to a third outlet 32, or second drain. This drain is in fluid communication with an apparatus operative to remove the salt wastes from the waste solution, such as by evaporation, filtration, chemical precipitation or other means.

Detailed Description Text (11):
The rinse/brine refill cycle 18 moves city water 50 from inlet 22 through resin vessel 24 thereby to rinse the resin of any remaining salt. The rinse water 60 is then sent from resin vessel 24 to brine tank 28 where it mixes with rock salt

provided by the user thereby to create brine for use in the next cycle.

Detailed Description Text (14):
With reference to FIG. 3(a), it may be seen that the brine draw cycle 14 includes pumping brine 54 from brine tank 28 with pump 34 through valve 1 and into resin vessel 24. Pump 34 is turned on by an electric micro switch that is activated as soon as the brine valve flapper opens in valve 1. Pump 34 may be a gear pump, self-priming piston/positive displacement pump, or other pump as known in the art. The flow rate of brine 54 is controlled by flow controller 44, which is preferably an orifice type flow controller as known in the industry. Clean water 56 displaced by brine 54 in resin vessel 24 is moved through valve 3A and out the second outlet, or first drain 30, which preferably connects to a sewer. During the brine draw cycle 14, valves 2, 3, 4, 5 and 6 remain closed.

Detailed Description Text (15):
With reference to FIG. 4(a), it may be seen that once resin vessel 24 has filled with brine 54, the waste draw cycle 16 begins, wherein valve 3A closes and valve 6 opens, while valve 1 remains open and pump 34 continues to pump. At this point, the solution in resin vessel 24 has undergone the ion-exchange process, such that a waste water solution 58 is formed that includes ions such as calcium and magnesium. This volume of solution is moved through valve 6 and out the third outlet, or second drain 32, to a collection, or processing, device operative to dispose of the waste solution 58, preferably an evaporation device. The continued pumping of pump 34 of brine 54 through valve 1 pushes the waste solution 58 through valve 6 and out second drain 32. The volume expelled through valve 6 and out second drain 32 may be from 0.25 to 2.0 bed volumes. A bed volume is the liquid equivalent of the volume of resin in the softener. Valves 2, 3, 3A, 4 and 5 remain closed during this cycle.

Detailed Description Text (16):
With reference to FIG. 5(a), it can be seen that once the waste solution resulting from regeneration of the ion-exchange resin is removed from the resin vessel, the rinse/brine refill cycle 18 begins by opening valves 3 and 1 and closing the remaining valves, thereby to allow city water 50 from inlet 22 to enter resin vessel 24 and move through valve 1 into brine tank 28 as rinse water 60. It should be noted that, while solution is moving through pump 34 in a direction counter to its pumping direction, pump 34 is not active. This step rinses the ion exchange resin 36 in resin vessel 24 as well as refills the solution in brine tank 28.

Detailed Description Text (19):
Manifold 110 is in fluid communication with the regenerant reservoir, or brine tank 28, and resin vessel 24. Pump 34 is in fluid communication between brine tank 28 and manifold 110 and operative to pump brine 54 from brine tank 28 to manifold 110. Manifold 110 includes valves 1, 2, 3, 3A, 4, 5 and 6 as shown.

Detailed Description Text (20):
Ion exchange resin 36 is disposed in resin vessel 24. Resin vessel 24 includes three concentrically disposed conduits, which define various inlets and outlets between resin vessel 24 and manifold 110. In particular, an outer diameter 42 of resin vessel 24 constitutes a first combination inlet/outlet between resin vessel 24 and manifold 110. This combined inlet/outlet may circulate water from inlet 22 into resin vessel 24 when valve 3 is open, and may circulate water to third outlet, or second drain, 32 from resin vessel 24 when valve 6 is open. The innermost concentric conduit, riser tube 38, constitutes a second combination inlet/outlet between manifold 110 and resin vessel 24. In particular, regenerant solution, or brine 54, may be circulated from regenerant reservoir, or brine tank 28, through riser tube 38 into resin vessel 24 when valve 1 is open and pump 34 is active. Water from inlet 22 may be circulated through resin vessel 24, into riser tube 38 and into brine tank 28 when valves 1 and 3 are open and pump 34 is inactive. Outer tube 40 within resin vessel 24 constitutes a resin vessel outlet in fluid

communication with second outlet, or first drain 30 of manifold 110.

Detailed Description Text (21):
The fluid communication of brine tank 28 with manifold 110 may be accomplished using a single conduit, as shown, or multiple conduits to the extent understood in the art. In particle, the single conduit fluid communication between brine tank 28 and manifold 110 as shown constitutes a third combination inlet/outlet of manifold 110 itself. That is, fluid can pass either from brine tank 28 to manifold 110 or in the reverse direction from manifold 110 to brine tank 28, depending upon the valve state of the valves in manifold 110 and the state of operation of pump 34.

Detailed Description Text (22):
It is preferred that ion exchange resin 36 be a resin with very fast kinetics. Preferred resins include those manufactured by Purolite, located in Bala Cynwyd, Pa., including the Purolite SST resins and the Purolite C-100-FM. These Purolite resins are classified as "Fine Mesh" resins and have relatively small diameter bead sizes that may range from approximately 16 US mesh to 70 US mesh. The Purolite SST resins, such as the SST-60, have fast kinetics because the ion exchange region is only on the surface of the bead, rather than throughout the sphere of the bead. Such resins are known in the industry as Shallow Shell or Shortened Diffusion Path (SDP) resins. The Purolite C100FM has fast kinetics due to very small bead size. It should be understood that the present invention contemplates the use of ion exchange resins having both standard and very fast kinetics, as well as ion exchange resins which are similar or equivalent to the Purolite versions.

Detailed Description Text (23):
As shown by arrows, during service cycle 12, city water 50 enters inlet 22, passes through open valve 3 and into resin vessel 24 where it contacts ion exchange resin 36. Softened water 52, resulting from the ion exchange reaction of city water 50 with ion exchange resin 36, then flows through riser tube 38 through open valve 4 and out outlet 26 to the home or industrial water supply outlets. Valves 1, 2, 3A, 5 and 6 remain closed.

Detailed Description Text (24):
FIG. 3(b) shows the brine draw cycle 14 during operation of the water softening apparatus 100. Here, brine 54 from brine tank 28 is pumped by pump 34 through open valve 1 into resin vessel 24 through the bottom of the riser tube 38. The flow rate of brine 54 is controlled by flow controller 44, which is preferably an orifice type flow controller known in the art. Clean water 56 displaced by brine 54 enters outer pipe 40 and passes through open valve 3A to drain 30. It is preferred that only an approximate volume of clean water 56 equal to the volume existing in the resin vessel 24 prior to brine 54 entering the resin vessel 24 should be expelled from drain 30, so as to prevent brine 54 from being expelled down drain 30 to the municipality waste treatment system. Valves 2, 3, 4, 5, and 6 remain closed during the brine draw cycle 14.

Detailed Description Text (25):
FIG. 4(b) shows the operation of the waste draw cycle 16 in apparatus 100. The solution in the resin vessel 24 now includes the waste solution 58 from regeneration of ion exchange resin 36 by contact with the brine 54 drawn into resin vessel 24 during the brine draw cycle 14. In the waste draw cycle 16, brine solution 54 continues to be drawn from brine tank 28 by pump 34 through open valve 1 and forced down riser tube 38 into resin vessel 24 and through ion exchange resin 36. This new solution displaces the regenerant waste solution 58, which is moved through the outer diameter 42 of resin vessel 24 through open valve 6 and out second drain 32, to a disposal device such as an evaporation device. Valves 2, 3, 3A, 4 and 5 remain closed.

Detailed Description Text (26):
FIG. 5(b) shows the rinse/brine refill cycle 18 in apparatus 100. City water 50

from inlet 22 passes through opened valve 3 into resin vessel 24 and through ion exchange resin 36. The rinse solution 60 passes through riser tube 38 and through open valve 1, further through pump 34 which is not active and into brine tank 28. The flow to brine tank 28 is controlled by flow controller 44, such as an orifice type flow controller. When brine tank 28 has reached its capacity, as determined by a time factor relating to the flow rate volume and tank capacity (or other known methods such as a float-check valve), the flapper in valve 1 is closed, thereby to stop the flow of rinse solution 60 to brine tank 28. To replenish the brine concentration in brine tank 28, a user adds rock salt 62 to the solution in brine tank 28. Brine tank 28 does not require agitation, rather the brine solution 54 saturates by soaking in the rock salt 62.

Detailed Description Text (27):
FIG. 6(b) shows the backwash cycle 20 in operation in apparatus 100. Here, city water 50 enters inlet 22 passes through open valves 2 and 4, down riser tube 38 and into resin vessel 24. The rinse water 60 passes through ion exchange resin 36 in a counter-flow direction thereby to fluff up the resin bed, and up outer pipe 40, through open valve 3A and out second outlet, or first drain 30. Backwash cycle 20 has the effect of fluffing up the beads of ion exchange resin 36, thereby to permit increased water flow through resin vessel 24. While not shown, it should also be appreciated that ion-exchange resin 36 may be further rinsed, if desired, by opening valves 3 and 5 so that city water 50 passes through inlet 22 and into resin vessel 24, up riser tube 38 and out second outlet, or first drain, 30.

Detailed Description Text (28):
Turning to FIG. 7, it may be seen that water softening system 200 according to the present invention includes a manifold, such as multi-port valve 110; resin vessel 24 containing ion-exchange resin 36; regenerant reservoir, such as brine tank 28; a processing device, such as evaporation device 64; a water source, such as city water line inlet 66; a water tap, such as a home or industry water line outlet 68 that provides water for consumption, and a water drain, such as sewer 70. "Consumption" should be understood in the general sense of any use to which softened water 52 might be put. Pump 34 is disposed in fluid communication between brine tank 28 and manifold 110.

Detailed Description Text (29):
City water 50 is passed from the water source, such as the city water line inlet 66, to inlet 22 in manifold 110. Softened water 52 passes from first outlet 26 of manifold 100 to the home or industry water line outlet 68 where it can be used in residential and industrial processes, such as bathing, washing, drinking, etc. Clean water 56 and rinse water 60 may be sent to the sewer 70 through second outlet, or first drain 30, of manifold 110. Clean water 56 and rinse water 60 should contain little or no salt waste, and thus are acceptable solutions for municipal waste treatment systems. Waste solution 58 is passed through third outlet, or second drain 32, of manifold 110 to a processing device, such as evaporation device 64. Regenerant solution, such as brine 54 is pumped from brine tank 28 to manifold 110 by pump 34. Rinse water 60 flows from manifold 110 to brine tank 28 to allow replenishment of brine 54 in brine tank 28.

CLAIMS:

1. A water softening apparatus adapted to be placed in fluid communication with a water drain, a processing device, a water source that provides water containing undesired ions, and a water tap that dispenses water for consumption, said water softening apparatus operative to remove the undesired ions from water processed thereby, comprising: (a) a resin vessel sized and adapted to receive a selected volume of a fluid, said resin vessel containing an ion-exchange resin that is capable of chemically shifting between an active state operative to exchange selected preferred ions therein for the undesired ions contained in the water when in contact therewith and an exhausted state operative to exchange the undesired

ions therein for the selected preferred ions contained in a regenerant solution when in contact therewith; (b) a regenerant reservoir adapted to receive the regenerant solution containing the selected preferred ions; and (c) a manifold in fluid communication with said resin vessel and said regenerant reservoir, said manifold having a first inlet in fluid communication with the water source, a first outlet in fluid communication with the water tap, a second outlet in fluid communication with the water drain, and a third outlet in fluid communication with the processing device, said manifold including a plurality of fluid pathways communicating between said inlet, said outlets, said resin vessel and said regenerant reservoir, and a plurality of valves associated with said fluid pathways that are configurable into a plurality of valve states whereby in a first valve state fluid can flow through said first inlet, through said resin vessel and through said first outlet, whereby in a second valve state fluid can flow from said regenerant reservoir through said resin vessel and through said second outlet, whereby in a third valve state fluid can flow from said regenerant reservoir through said resin vessel and through said third outlet, and whereby in a fourth valve state fluid can flow through said first inlet, through said resin vessel and into said regenerant reservoir.

10. A water softening apparatus according to claim 1 wherein said ion-exchange resin is a shallow shell/shortened diffusion path resin.

11. A water softening apparatus according to claim 1 wherein said ion-exchange resin is a fine mesh resin having a bead diameter of approximately 16-70 mesh.

12. A water softening system, comprising: (a) a water source that provides water containing undesired ions; (b) a water tap that dispenses water for consumption; (c) a water drain; (d) a processing device; (e) a regenerant reservoir that is sized and adapted to receive a selected volume of a regenerant solution containing selected preferred ions; (f) a resin vessel sized and adapted to receive a selected volume of a fluid; (g) an ion-exchange resin disposed in said resin vessel, said ion-exchange resin capable of chemically shifting between an active state operative to exchange the selected preferred ions therein for the undesired ions contained in the water when in contact therewith and an exhausted state operative to exchange the undesired ions therein for the selected preferred ions contained in the regenerant solution when in contact therewith; and (h) a manifold in fluid communication with said resin vessel and said regenerant reservoir, said manifold having a first inlet in fluid communication with said water source, a first outlet in fluid communication with said water tap, a second outlet in fluid communication with said water drain, and a third outlet in fluid communication with said processing device, said manifold including a plurality of fluid pathways communicating between said inlet, said outlets, said resin vessel and said regenerant reservoir, and a plurality of valves associated with said fluid pathways that are configurable into a plurality of valve states whereby in a first valve state water from said water source flows through said first inlet, through said resin vessel and through said first outlet to said water tap when said ion-exchange resin is in the active state thereby to remove the undesired ions from the water, whereby in a second valve state regenerant solution flows from said regenerant reservoir into said resin vessel and water flows from said resin vessel through said second outlet to said water drain, whereby in a third valve state regenerant solution flows from said regenerant reservoir into said resin vessel when said ion-exchange resin is shifted toward the exhausted state thereby to shift the ion-exchange resin toward the active state and form a waste solution that flows through said third outlet to said processing device, and whereby in a fourth valve state water from said water source flows through said first inlet, through said resin vessel and into said regenerant reservoir thereby to replenish the volume of fluid therein.

15. A water softening system according to claim 12 wherein the regenerant solution is a brine solution.

17. A method for softening water that contains undesired ions, comprising: (a) providing an ion-exchange resin in a resin vessel sized and adapted to receive a fluid, wherein said ion-exchange resin is capable of chemically shifting between an active state operative to exchange selected preferred ions therein for the undesired ions contained in the water when in contact therewith and an exhausted state operative to exchange the undesired ions therein for the selected preferred ions contained in a regenerant solution when in contact therewith; (b) contacting said ion-exchange resin with the water that contains the undesired ions when said ion-exchange resin is shifted toward the active state, thereby to remove the undesired ions from the water and shift said ion-exchange resin toward the exhausted state; (c) contacting said ion-exchange resin with the regenerant solution containing the selected preferred ions when said ion-exchange resin is shifted toward the exhausted state, thereby to remove the preferred ions from the regenerant solution so as to shift said ion-exchange resin toward the active state; (d) forming a waste solution containing the undesired ions; (e) displacing a selected volume of water in said resin vessel with said regenerate solution and passing the selected volume of water to a water drain; (f) disposing the waste solution containing the undesired ions via a processing device that is separate from a drainage line; and (g) rinsing said ion-exchange resin with water thereby to form a rinse solution and thereafter transporting the rinse solution to a regenerant reservoir.

20. A method according to claim 17 wherein the step of contacting said ion-exchange resin with the regenerant solution includes contacting said ion-exchange resin with between 0.25 and 2.0 bed volumes of the regenerant solution.

21. A method according to claim 17 wherein the step of contacting said ion-exchange resin with the regenerant solution includes transporting the regenerant solution from a regenerant reservoir into said resin vessel.

24. A method according to claim 17 including the step of adding rock salt to said regenerant reservoir, thereby to form a brine solution from said rinse solution.

25. A method for softening hard water using a water softening apparatus that includes an ion-exchange resin disposed in a resin vessel that is sized and adapted to receive a selected volume of a fluid, wherein said ion-exchange resin is one that is operative to soften hard water, the method comprising: (a) displacing a quantity of water in said resin vessel with a regenerant solution, wherein said regenerant solution is operative to regenerate said ion-exchange resin when said ion-exchange resin is exhausted and thereby form in said resin vessel a waste solution having salt contaminants therein; (b) transporting said displaced water to a water drain adapted to receive water for disposal; (c) displacing a quantity of waste solution in said resin vessel with said regenerant solution thereby to fill said resin vessel with said regenerant solution; (d) transporting said displaced waste solution to a processing device operative to process a solution having salt contaminants therein; (e) displacing a quantity of regenerant solution in said resin vessel with water thereby to rinse said ion-exchange resin and form a rinse solution in said resin vessel; (f) transporting said displaced regenerant solution to a regenerant reservoir adapted to receive said regenerant solution; (g) displacing a quantity of rinse solution in said resin vessel with water; (h) transporting said displaced rinse solution to at least one of said regenerant reservoir and said water drain; (i) flowing hard water through said resin vessel thereby to contact said hard water with said ion-exchange resin to form softened water; and (j) transporting said softened water to a water tap operative to dispense said softened water for consumption.

26. A water softening apparatus adapted to be placed in fluid communication with a water drain, a processing device, a water source that provides water containing undesired ions, and a water tap that dispenses water for consumption, said water

softening apparatus operative to remove the undesired ions from water processed thereby, comprising: (a) a resin vessel sized and adapted to receive a selected volume of a fluid, said resin vessel having an upper portion and a lower portion, said lower portion containing a selected amount of an ion-exchange resin; (b) a regenerant reservoir adapted to receive a selected volume of a fluid; and (c) a manifold in fluid communication with said resin vessel and said regenerant reservoir, said manifold having: (1) a first inlet adapted to be placed in fluid communication with the water source; (2) a first outlet adapted to be placed in fluid communication with the water tap; (3) a second outlet adapted to be placed in fluid communication with the water drain; (4) a third outlet adapted to be placed in fluid communication with the processing device; (5) a first conduit fluidly communicating between said first inlet, said third outlet, and said upper portion of said resin vessel; (6) a second conduit separate from said first conduit and fluidly communicating between said second outlet and said upper portion of said resin vessel; (7) a third conduit separate from said first and second conduits and fluidly communicating between said first outlet, said second outlet, said regenerant reservoir and said lower portion of said resin vessel; (8) a plurality of valves disposed in said conduits, wherein said valves are configurable into a plurality of valve states (A) whereby in a first valve state fluid can flow from said first inlet, through said first conduit into said upper portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in a downflow direction, into said third conduit and out said first outlet; (B) whereby in a second valve state fluid can flow from said regenerant reservoir, through said third conduit into said lower portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in an upflow direction, through said second conduit and out said second outlet; (C) whereby in a third valve state fluid can flow from said regenerant reservoir, through said third conduit into said lower portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in an upflow direction, through said first conduit and out said third outlet; and (D) whereby in a fourth valve state fluid can flow from said first inlet, through said first conduit into said upper portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in a downflow direction, through said third conduit and into said regenerant reservoir.

27. A water softening apparatus adapted to be placed in fluid communication with a water drain, a processing device, a water source that provides water containing undesired ions, and a water tap that dispenses water for consumption, said water softening apparatus operative to remove the undesired ions from water processed thereby, comprising: (a) a resin vessel sized and adapted to receive a selected volume of a fluid, said resin vessel having an upper portion and a lower portion, said lower portion containing a selected amount of an ion-exchange resin, wherein said resin vessel includes a mouth in said upper portion of said resin vessel, a first tube extending through said mouth and opening into said lower portion of said resin vessel, and a second tube extending through said mouth and opening into said upper portion of said resin vessel; (b) a regenerant reservoir adapted to receive a selected volume of a fluid; and (c) a manifold in fluid communication with said resin vessel and said regenerant reservoir, said manifold having: (1) a first inlet adapted to be placed in fluid communication with the water source; (2) a first outlet adapted to be placed in fluid communication with the water tap; (3) a second outlet adapted to be placed in fluid communication with the water drain; (4) a third outlet adapted to be placed in fluid communication with the processing device; (5) a first conduit fluidly communicating between said first inlet, said third outlet, and said mouth of said resin vessel; (6) a second conduit separate from said first conduit and fluidly communicating between said second outlet and said second tube of said resin vessel; (7) a third conduit separate from said first and second conduits and fluidly communicating between said first outlet, said second outlet, said regenerant reservoir and said first tube of said resin vessel; (8) a plurality of valves disposed in said conduits, wherein said valves are configurable into a plurality of valve states (A) whereby in a first valve state

fluid can flow from said first inlet, through said first conduit and through said mouth into said upper portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in a downflow direction, through said first tube and into said third conduit and out said first outlet; (B) whereby in a second valve state fluid can flow from said regenerant reservoir, through said third conduit and through said first tube into said lower portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in an upflow direction, through said second tube and into said second conduit and out said second outlet; (C) whereby in a third valve state fluid can flow from said regenerant reservoir, through said third conduit and through said first tube into said lower portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in an upflow direction, through said mouth and into said first conduit and out said third outlet; and (D) whereby in a fourth valve state fluid can flow from said first inlet, through said first conduit and through said mouth into said upper portion of said resin vessel, through said ion-exchange resin in said lower portion of said resin vessel in a downflow direction, through said first tube and through said third conduit and into said regenerant reservoir.

29. A method for softening water that contains undesired ions, comprising: (a) providing an ion-exchange resin in a resin vessel sized and adapted to receive a fluid, wherein said ion-exchange resin is capable of chemically shifting between an active state operative to exchange selected preferred ions therein for the undesired ions contained in the water when in contact therewith and an exhausted state operative to exchange the undesired ions therein for the selected preferred ions contained in a regenerant solution when in contact therewith; (b) contacting said ion-exchange resin with the water that contains the undesired ions when said ion-exchange resin is shifted toward the active state, thereby to remove the undesired ions from the water and shift said ion-exchange resin toward the exhausted state; (c) contacting said ion-exchange resin with the regenerant solution containing the selected preferred ions when said ion-exchange resin is shifted toward the exhausted state, thereby to remove the preferred ions from the regenerant solution so as to shift said ion-exchange resin toward the active state; (d) forming a waste solution containing the undesired ions; (e) displacing a selected volume of water in said resin vessel with said regenerant solution and passing the selected volume of water to a water drain; (f) disposing the waste solution containing the undesired ions via a processing device that is separate from a drainage line; (g) displacing the regenerant solution in said resin vessel with water thereby to rinse said ion-exchange resin and form a rinse solution in said resin vessel; (h) transporting said rinse solution to a regenerant reservoir; and (i) adding rock salt to said regenerant reservoir, thereby to form a brine solution from said rinse solution.

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